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FIBER REINFORCED METALLIC COMPOSITES

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One of the more unconventional methods to provide high strength, light weight materials for application over a wide range of temperature is the utilization of composites containing fibers. In such composites a combination of metals or ceramics is incorporated in such a manner that the desirable properties of each of the constituents is utilized. Two types of metallic fibers are available; high strength wire, and whiskers. Whiskers have been reported to have tensile strengths approaching the theoretical strength of metals.⁽¹⁾ If a major portion or all of this strength can be utilized in an engineering material a superior product could be obtained. By their very nature, however, whiskers are difficult to produce, handle, and fabricate. Because of this difficulty much must be learned about how to handle such fine fibers before they can be used successfully and it has been expedient to conduct preliminary experiments using wire. These polycrystalline materials have been available for many years, and more recently, ceramics have also been produced in filament form. Since metallic filaments exhibit unusually high tensile properties and can be drawn to 1 mil diameter or less they may be used as substitutes for whiskers to study the mechanisms by which bonding may be accomplished. Furthermore, such effects as fiber orientation, length, and stability can be studied using finely drawn wires. Ultimately the background obtained may be utilized in the fabrication of composites containing whiskers. Meanwhile, the high strength metal or ceramic filaments which are now available may be used for a great many composites.

Although considerable work has been done on the reinforcement of plastics by the utilization of high strength glass filaments, relatively little has been published on the reinforcement of metals with stronger metal fibers. This is not to say that such composites have been completely ignored. For example: an Italian patent issued in 1948 covers the reinforcement of aluminum and bronze utilizing steel wool as the reinforcing media.⁽²⁾ Many organizations are interested or actually conducting work in this field, although the only known published data on strengths of reinforced metals containing metal fibers is that of the Clevite Corporation.⁽³⁾ Others at Armour Research Foundation,⁽⁴⁾ Ohio State,⁽⁵⁾ and Alfred University,⁽⁶⁾ to name a few, have done work on the reinforcement of ceramics using metallic filaments. This paper summarizes some of the work conducted at the Clevite Research Center and work now in progress at the Lewis Research Center of the NASA.

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First, the work conducted at Clevite under Navy sponsorship will be examined. The results obtained from these studies show some of the significant increases in strength obtained by reinforcing titanium and a titanium alloy with molybdenum fibers. At the time the work was done, it

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was felt that a maximum density of 0.26 pounds per cubic inch was desired. This density was selected so that the product would be light weight and predominately titanium. To keep the overall density within the desired limits it was not possible to utilize more than 50 volume percent molybdenum fiber.

Molybdenum wire was chosen as the reinforcement because of its high strength, availability, and because molybdenum forms a continuous series of solid solutions with titanium. In other words no formation of brittle intermetallic compounds would be possible at the fiber matrix interface.

Specimens were prepared using powder metallurgical techniques. Titanium alloy powder of -100 mesh was made by hydriding Ti-6Al-4V alloy sheet which was subsequently crushed and ball milled. The powder obtained from ball milling was then vacuum outgassed to remove the hydrogen and dry blended with 10 mil molybdenum wire which had been cut to lengths of $1/10$ to $1/4$ inch. The blend was cold pressed into billets, and vacuum sintered for one hour at 1800° F to remove the remaining hydrogen and to partially density the billet. The $1\frac{1}{2}$ inch diameter billets were canned in mild steel and extruded to $5/8$ inch rod (extrusion ratio $\approx 6:1$) at 2100° F. These rods were hot rolled in grooved rod rolls with initial rolling done at 1800° F and, as rolling progressed, the temperature was reduced until at $3/16$ inch diameter the rolling temperature was 1450° F. The steel jacket was removed and the rod was given a final cold pass to improve the surface and reduce the rod to $1/8$ inch diameter. The rods were annealed for 2 hours at 1350° F. Control specimens of unreinforced alloy were run at the same time using the same base materials and methods.

Figure 1 shows what happened to the wire during the working operations. Originally, the filaments were randomly oriented within the billet. During extrusion, the wires, particularly those at the outer surface of the rod, began to orient themselves parallel to the direction of working. Rolling increased this preferred orientation and the resulting rod consisted of wires oriented parallel to each other and parallel to the long axis of the rod. In addition to the change in orientation, the filaments were reduced in diameter from the original 10 mils to 2 mils with a corresponding increase in length. The filament length could not be measured because the wires dipped beneath the surface of the metallographic plane. Most of the wires were estimated to be one third to one half the length of the specimen although a few may have extended completely through the specimen. The individual wires retained their identity and a slight diffusion zone could be seen at the wire matrix interface.

The results of tensile tests conducted on composites composed of the Ti alloy reinforced with 20 volume percent molybdenum fiber are shown in Figure 2. These tests were conducted at room temperature, 1000° , 1200° , and 1400° F. The improvement in strength of the composites over that of the alloy rod produced from the same powder is evident. At room temperature the improvement was approximately 30,000 psi and an advantage was maintained at elevated temperature as shown in figure 2.

Figure 3 shows the same data plotted as the tensile strength - density ratio versus temperature. While at room temperature the unreinforced

alloy had a higher tensile-strength to density ratio than the composite, the advantage was lost as the temperature increased. Although the curves show that the advantage persisted to 300° F the actual cross-over point is open to some question because of the lack of data at the temperatures between room temperature and 1000° F.

Improvements in other properties of the titanium alloy were also noted when reinforcing fiber was used. Figure 4 shows the results of a series of tests in which the modulus of elasticity was measured at various temperatures using dynamic methods. Of particular interest is the fact that beyond 600° F the modulus of the titanium alloy dropped off quite rapidly while at temperatures up to 1400° F the composites exhibited no such drop off. It should also be pointed out that the modulus of elasticity increased with increasing fiber content. These results demonstrate that by simply varying the amount of fiber it was possible to produce a "tailor made" composite having any desired modulus of elasticity, i.e., between 16×10^6 psi and 50×10^6 psi.

For materials of this type it is also necessary to know the stress rupture properties as well as the short time tensile strength. The composite composed of the titanium alloy reinforced with 20 volume percent wire was tested at 1200° F for stress rupture life. The results of these tests did not show the degree of success observed in the tensile tests, however, there was reason to believe that the poor stress rupture life was due to oxidation of the surface filaments.

A group of specimens composed of unalloyed titanium reinforced with molybdenum wire was also tested for stress rupture life at a lower temperature where the molybdenum fibers would not oxidize and sublime. The composites used for these tests were produced in exactly the same manner as previously described except that unalloyed titanium was used as the matrix. Figure 5 shows the results of these tests. At 800° F unalloyed titanium had a 100 hour rupture life at 20,000 psi. The incorporation of 10 volume percent reinforcement increased the life 10 fold to 1000 hours. At 1000° F and under the same stress unreinforced material had a life of 0.1 hour while the composite had a life of 100 hours. Thus, the addition of reinforcing filaments had a marked effect on the stress rupture properties.

Some of the results and conclusions that may be obtained from an analysis of the data obtained on reinforced titanium composites may be summarized as follows. First, it was definitely shown that the tensile strength of a titanium alloy was significantly improved by the addition of molybdenum fibers and these improvements were noted over a wide range of temperatures. It was also found that the strength to density ratio of the titanium alloy could be improved significantly, particularly at elevated temperatures by reinforcing with molybdenum fibers. Second, it was shown that the modulus of elasticity of the titanium alloy - molybdenum composite was increased proportionately as the quantity of molybdenum increased up to 40 volume percent fiber. Third, unalloyed titanium, reinforced with molybdenum fibers, was greatly improved from a stress rupture standpoint at test temperatures below the temperature at which molybdenum oxidizes.

material and tungsten as the fiber. These materials were chosen because of their mutual insolubility. Also, copper was used because, when molten, it wets tungsten. Furthermore, after exposure to temperatures slightly above the melting point of copper, the room temperature properties of the tungsten fiber were not seriously effected.

Figure 8 illustrates the steps involved in producing the tungsten reinforced copper bundles. Cut lengths of tungsten wire were cleaned with sodium peroxide and ammonium hydroxide and loaded into an alundum tube. This tube was then placed in a closed end quartz tube having a slug of copper infiltrant in the bottom. The entire assembly was heated to 2200° F and held for one hour at temperature. During infiltration, the spaces between the wires of the tightly packed bundles served as capillary tubes through which the molten copper could flow. The specimens were kept under a vacuum during infiltration to prevent oxidation of the tungsten and thereby provided a clean wire surface. This was essential since it was found that any surface film on the wire greatly reduced the chances of producing a successful infiltration.

Some variation in this procedure was found to be necessary when specimens of low fiber content were made. Because of the larger openings between wires, capillary rise could not take place and it became necessary to "top feed" the specimens by placing the infiltrating material in the tube above the wires and allowing gravity flow to take place. The specimens ranged from 40 mils to 1/8 inch in diameter and from three to six inches in length.

Figure 9 shows a cross section of a test specimen and illustrates the packing of the wires within the composite. The standard procedure for determining the relative amount of copper and tungsten was to section the specimen after testing and measure the cross-sectional area of the specimen by planimentering a photo of the cross section. This area was used as the basis for tensile strength calculations. The wires in the cross section were then counted to determine the volume percent copper and tungsten.

Specimens of copper-tungsten fiber composites such as those described were tensile tested at room temperature. Experimental data has been obtained to date that shows the strength of composites ranging in fiber content from 14 to 77 volume percent. As a base line for comparison a revised strength-composition diagram was developed. Figure 10 shows this strength-composition curve (represented by the solid line) obtained by refining the calculations used to plot the curve in figure 6. This revised curve may be divided into two sections. The first portion of the curve represents a composition range in which copper would be the major load carrying constituent. This range can be calculated (see appendix) to extend from 0 to about 5.3 volume percent fiber. In composites containing less than 5.3 percent, failure of tungsten fibers would not immediately result in failure of the composite. In fact, since copper would be carrying the bulk of the load, the fibers would fracture when the copper was elongated, as little as 1.5 percent which was the elongation of the tungsten fibers at their fracture stress. Immediately after the fibers fracture, the load previously carried by the fibers would be transmitted to the copper. The copper would then elongate until the ultimate strength of the copper would be reached. The elongation at this point could be as much as 40 percent.

The approach used at the Lewis Research Center has been to study the mechanisms by which fibers strengthen composites. The specific objectives of the research program were to determine whether in composites of mutually insoluble metallic materials, fibers could be combined in all proportions with the matrix and whether each constituent would carry its proportional load. The second objective was to utilize short length, axially oriented fibers, and to determine the strength of such composites compared with both the calculated strength and actual strength of composites made with continuous reinforcing fibers.

To furnish a basis for these studies it was felt that the problem should be approached by calculating the strengths of composites containing varying proportions of fiber and matrix. Experimental values could then be compared to the calculated values.

As a first approximation, it was assumed that each constituent in a two component composite would carry a load proportional to the tensile strength of the constituent and the volume of the constituent present; then the tensile strength of the composite would be equal to the tensile strength of the fiber multiplied by the volume percent of the fibers plus the tensile strength of the matrix times the volume percent of the matrix. Figure 6 shows a strength composition diagram for such a system based on these assumptions. The line drawn between the two constituents will give the strength of any composite of a given composition. For any specific composition, one might test specimens over a range of temperatures. Calculations of strength of the specific composite could be made if the strengths of the individual constituents at the different temperatures are known.

Replotting some of the data obtained for the titanium alloy reinforced with molybdenum will show, to some degree, how known metallurgical variables can account for differences between the experimental data and the calculated strength. Since the experiments were designed to obtain high strength composites rather than to study failure mechanisms, only portions of the data may be utilized to make the comparison. For this comparison the results obtained for the composite containing 20 volume percent fiber were used. Figure 7 shows a calculated curve of tensile strength versus temperature and the curve showing the experimental results.

Notice that the experimental data is well above the calculated data. In fact it is 10 to 15,000 psi higher. The calculations were made utilizing the strength of the original molybdenum fibers which were 10 mils in diameter. The strength of the alloy was known from experimental data. The reason for the difference between the experimentally determined curve relative to the calculated curve may be twofold. First, the titanium and the molybdenum in the composite are mutually soluble in each other and there may have been some solution strengthening effects due to alloying. Second, and perhaps of more significance, the tensile strength of the molybdenum fibers was probably higher after the product was extruded and rolled. Since the diameter of the molybdenum wires was reduced from 10 to 2 mils, an increase in their tensile strength may have been realized.

In the first studies at NASA, bundles of fibers cut to the length of the specimens to be tested were used. Copper was selected as the matrix

In the composition ranges above 5.3 volume percent, the tungsten fibers would become the predominant load carriers. Failure of some of the fibers would result in almost immediate failure of the composite. This may be reasoned as follows: As a load is applied to the composite, the tungsten fibers would carry the bulk of the load. Upon fracture of one or more fibers, the load would have to be supported by the remaining area of the composite. This effectively would increase the unit stress on all remaining fibers and the remaining matrix. Essentially immediate fracturing of the remaining fibers and matrix would be expected. The strain of the composite at maximum stress reached during testing the composite, would be essentially the same as that of the tungsten fibers. It was previously mentioned that the tungsten fibers have very low elongations at fracture, approximately 1.5 percent; whereas pure annealed copper has an elongation of about 35 percent at its ultimate tensile strength. If the copper in a composite were elongated to only 1.5 percent, it would be stressed well below its ultimate tensile strength. From a stress-strain diagram for copper, a stress of 8000 psi may be observed to occur at 1.5 percent elongation. The portion of the curve about 5.3 percent fibers, would then be a straight line having a strength intercept at 100 percent tungsten equal to the strength of the fiber and an extrapolated intercept, at 100 percent copper, equal to 8000 psi.

At extremely high tungsten fiber contents(perhaps at greater than 90 volume percent) there is a possibility that the slope of experimentally determined strength composition curves might increase due to triaxial strengthening of the thin copper films. At these extremely high fiber contents the interfiber spacing would be very small and the thin copper films between the fibers may be retarded from slipping. This blocking of slip might result in an increase in the strength of the composite somewhat above the calculated values.

Figure 10 also shows the results of tensile tests conducted at room temperature on tungsten-copper composites of various compositions. Tests were conducted on bundles containing continuous wires of three, five, and seven mil diameter and, although the test series is not complete, the composites containing the finer wires were, generally, stronger than the composites containing the larger wires. This was expected due to the fact that the finer wires had a higher initial tensile strength. Of greater interest, however, is the fact that the strength of the experimental composites, in the range of 14 to 77 volume percent-fiber, falls along the straight line previously calculated. These data show that, within the range of compositions studied, the strength of the composite is directly proportional to the amount of reinforcement present.

Future work on composite bundles of this type will be concerned with the variations which may be imparted to this curve due to closely packed fibers of different sizes in high fiber compositions, alloying between the fiber and matrix, and the effects on the strength of the composites when the filaments are electro polished to remove surface imperfections.

Figure 11 shows an interesting side light resulting from research on these composites. It was found that a composite body could be bent quite severely without fracture while a massive piece of tungsten of the same size shattered when it was bent approximately 30° on a two inch radius.

The second series of studies at the NASA was conducted to investigate the effects of fiber length on the tensile strength of composites. Discontinuous fiber composites were made by packing cut lengths of 5 mil tungsten wire in an alundum tube. The wires ranged in length from $1/8$ to $5/8$ inches although the majority were about $3/8$ inches long. Because the diameter of the tube was small in comparison to the fiber length, the fibers packed in such a manner that their long axis was oriented parallel to the long axis of the specimen.

The wires were then infiltrated with copper using top feeding in a manner comparable to that described for the bundles containing full length fibers. The minimum test section length of the specimens was $1\frac{1}{2}$ inches and obviously since the fibers were considerably shorter than the test section, the tests reflected the true effect of short fibers in a composite.

The results of a limited series of tensile tests conducted on these specimens are shown in figure 12. Specimens containing greater than 40 volume percent wire have not been tested. The data points shown on this curve represent the work done to date and it is possible that future data may modify some of this analyses. However, since all of the fiber contents tested in this series are relatively low, the mechanisms that will be discussed will apply to all of the specimens of this type. The figure again contains the straight line drawn between the ultimate tensile strength of the tungsten fibers and the strength previously utilized for the copper matrix. Note that most of the data points fall very close to this straight line. It may be recalled that the data for full length fibers fell almost exactly on the same straight line. Thus the strengths of the discontinuous fiber composites are almost identical to the strength of the specimens with the full length fibers. Actually the fact that the strengths obtained in the short length specimens fell upon the calculated curve corroborates metallographic evidence that the fibers and matrix failed in tension.

In order to explain the mechanism by which the discontinuous specimens achieved their strengths, a simplified model (fig. 13) of such a composite should be considered. Fibers in this type of composite were aligned parallel to each other and parallel to the long axis of the rod. Also, one may presume that the fibers overlapped each other by varying amounts and that they were individually bonded to the matrix. Because of the overlapping of these fibers, the stress was believed to be transferred from one fiber to the next by a shear mechanism. The actual shear strength of the copper to tungsten bond was not known. However, the surface area of the fine filaments is very high and, therefore, it is possible to transmit a considerable load from the matrix to the fiber or vice versa before the bond fails in shear.

The equations below show the procedure used to calculate a minimum bond length that would transmit a load to the fiber equal to the tensile strength of the fiber. To do this, the shear load was equated to the tensile load.

$$\text{Shear Load} = \text{Tensile Load}$$

$$A_s \times \sigma_s = A_T \times \sigma_T \quad (1)$$

$$2\pi rL \times \sigma_s = \pi r^2 \times \sigma_T \quad (2)$$

$$L = \frac{r}{2} \left(\frac{\sigma_T}{\sigma_s} \right) \quad (3)$$

$$\frac{\sigma_T}{\sigma_s} = \frac{330,000}{5,000} \quad (4)$$

$$L \geq 33r \quad (5)$$

In equation (1) the shear area of the specimen A_s multiplied by the shear strength of the matrix σ_s - was equated to the cross-sectional area of the fiber A_T times the tensile strength of the fiber σ_T . In equation (2) the circumference of the wire was multiplied by the length of the shear bond L and the shear stress and equated to the cross-sectional area of the fiber times the tensile strength of the fiber. This simplified to equation (3) which expresses the length of the required bond as $1/2$ the radius r times the ratio of the tensile strength of the fiber to the shear strength of the matrix. The tensile strength of the fiber was known to be of the order of 330,000 pounds per square inch. The shear strength of the copper, however, was obtained on the basis of several assumptions. The published shear strength of copper was found to be approximately 20,000 pounds per square inch. However, as mentioned previously the elongation of the composite was limited by the elongation of the tungsten. Therefore, using an analogy similar to that used for the strength of copper in the continuous composites, a conservative value was selected for the shear strength of copper. The value chosen for the shear strength of copper was 5000 pounds per square inch. If this is the case then equation (5) shows that the bond length required for tensile failure to be equal to or greater than 33 times the radius. Since most of the work done with the discontinuous fibers involved fibers 5 mils in diameter, the minimum length of copper tungsten bond necessary to cause a fiber to fail in tensile was thus found to be 0.082 inch. This was considerably less than the length of fibers (approximately $3/8$ inches) used for these types of specimens. Since the lengths used were more than adequate to obtain sufficient bonding the specimens failed in tension. The tensile strength data for composites made with short length fibers coincided with that obtained for continuous fibers. Metallographic examinations of the fractured specimens, particularly the longitudinal sections of the specimens, revealed that the tungsten fibers necked down within the specimens and broke at different elevations relative to the fracture surface. This supports the conclusions that had been drawn based on the analyses of the data and the calculations just made.

Implications of the results obtained from the discontinuous specimens are of considerable significance. First of all, one might produce composites utilizing short length fibers, more readily, in many cases, than composites containing long length fibers. In other words, the production of short length fiber composites is easier and faster and the process itself seems to have more commercial potential. Secondly, fiber composites

of varying properties may be produced by the simple expedient of varying the fiber content of the composite. Thirdly, since composites may be made of short length fibers and essentially have a strength equivalent to composites made from long fibers, it is conceivable that high strength composites can be made utilizing the same techniques, but using whiskers as the fibers. This latter conclusion has obviously the most far reaching significance for one may ultimately be able to tap the unusually high strength that whiskers are known to have particularly if the properties of the whiskers are not destroyed by the process of fabrication of the composite.

Briefly, then, the results of the investigations can be summarized as follows: It was possible to produce fiber reinforced composites by both powder metallurgy methods and liquid phase sintering. Furthermore, it has been shown that composites having higher room temperature and elevated temperature tensile strength than the matrix material can be made using reinforcing fibers. Reinforcing fibers also had a marked effect on the stress-rupture life of the matrix. The studies of fracture mechanisms have shown that in both continuous and discontinuous fiber composites consisting of mutually insoluble materials, the tensile strength was directly proportional to the amount of reinforcement present for a wide range of fiber to matrix compositions. Of even more importance, discontinuous fiber composites can be produced which have tensile strengths equal to the tensile strengths observed in continuous composites. The experience gained, then, may eventually lead to the practical utilization of some of the phenomenal strengths observed in whiskers.

APPENDIX - CALCULATED MAXIMUM FIBER CONTENT FOR COMPOSITE

IN WHICH COPPER IS THE PREDOMINANT LOAD CARRIER

As mentioned in the text, the strength-composition curve for these tungsten-copper composites may be divided into two areas: The area where tungsten governs the failure mechanism and carries the bulk of the load, and the area where copper is the predominant load carrier.

In the composition range where tungsten is the major load carrier, the load carried by the composite may be determined for any value of A_w by equation (1).

$$P = \sigma_w(A_w) + \sigma_{Cu_1}(A_{Cu}) \quad (1)$$

where

P = total load on the composite (pounds)

σ_w = tensile strength of tungsten (330,000 psi)

A_w = percent of cross-sectional area of composite occupied by tungsten

σ_{Cu_1} = tensile stress on copper at maximum elongation of the copper
(8000 psi)

A_{Cu} = percent of cross-sectional area of composite occupied by

copper = $100 - A_w$ (since $A_{Cu} + A_w = 100$ percent)

The maximum load carried by the composite, where copper is the predominant load carrier, after the fracture of any wires present, is determined by equation (2).

$$P = \sigma_{Cu_2}(A_{Cu}) \quad (2)$$

where

σ_{Cu_2} = the ultimate tensile strength of copper (26,500 psi)

The composition at which the predominant load carrying constituent changes from the copper matrix to the tungsten fibers may be calculated by equating (1) to (2) and solving for A_w . A value of 5.3 volume percent fiber was obtained for A_w .

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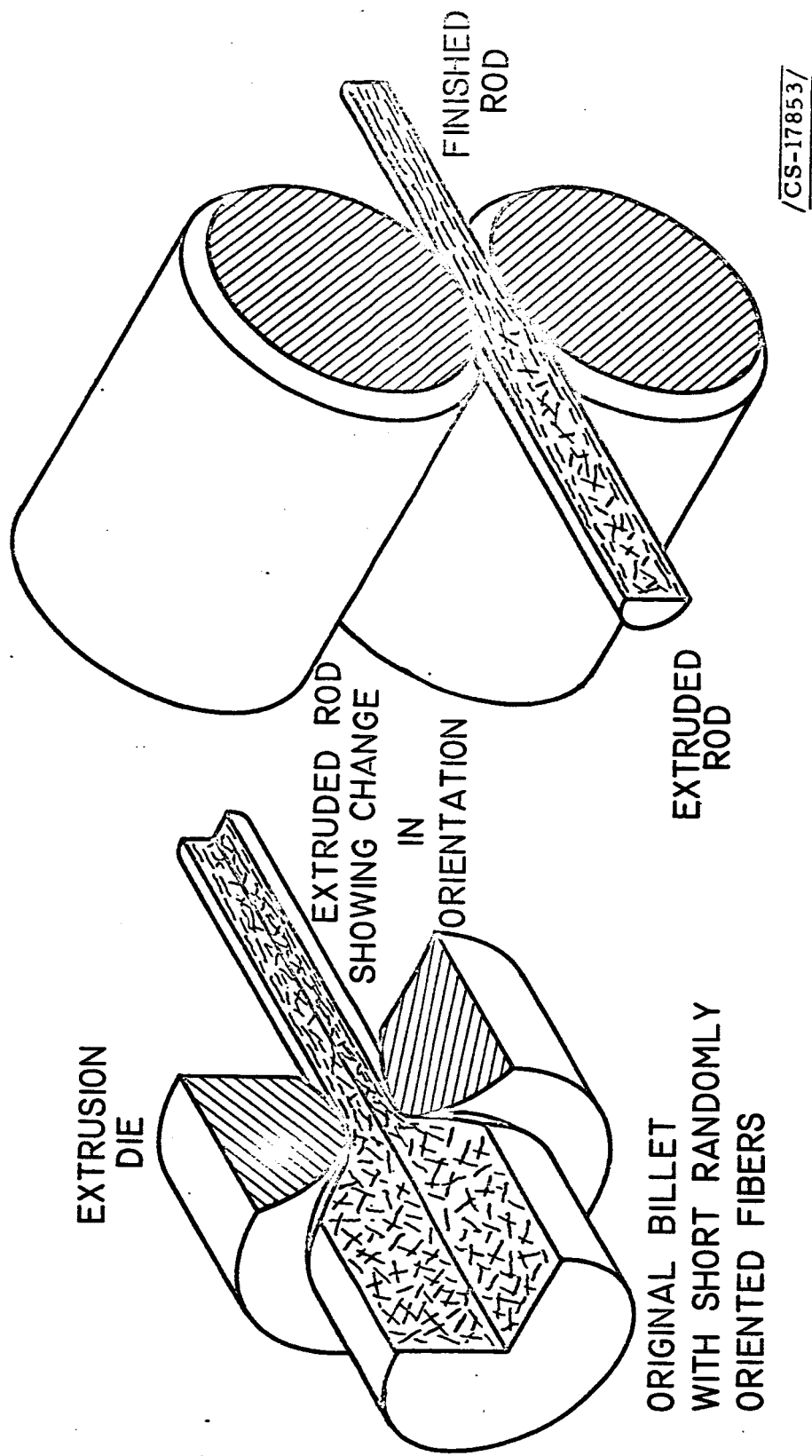


Figure 1. - Fabrication of discontinuous molybdenum fiber reinforced titanium composites.

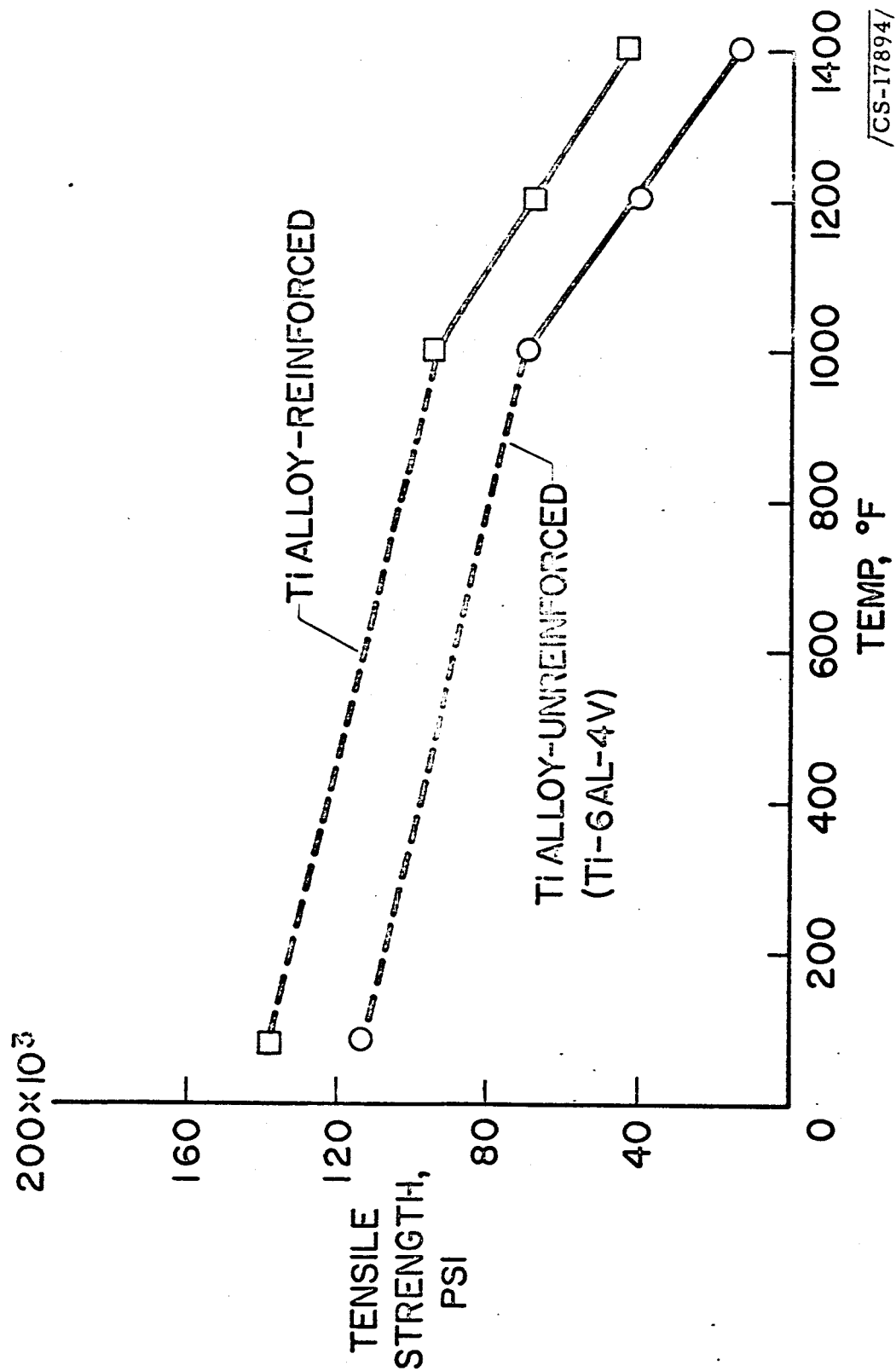


Figure 2. - Tensile strength versus temperature for unreinforced titanium alloy and alloy reinforced with 20 volume percent molybdenum fiber.

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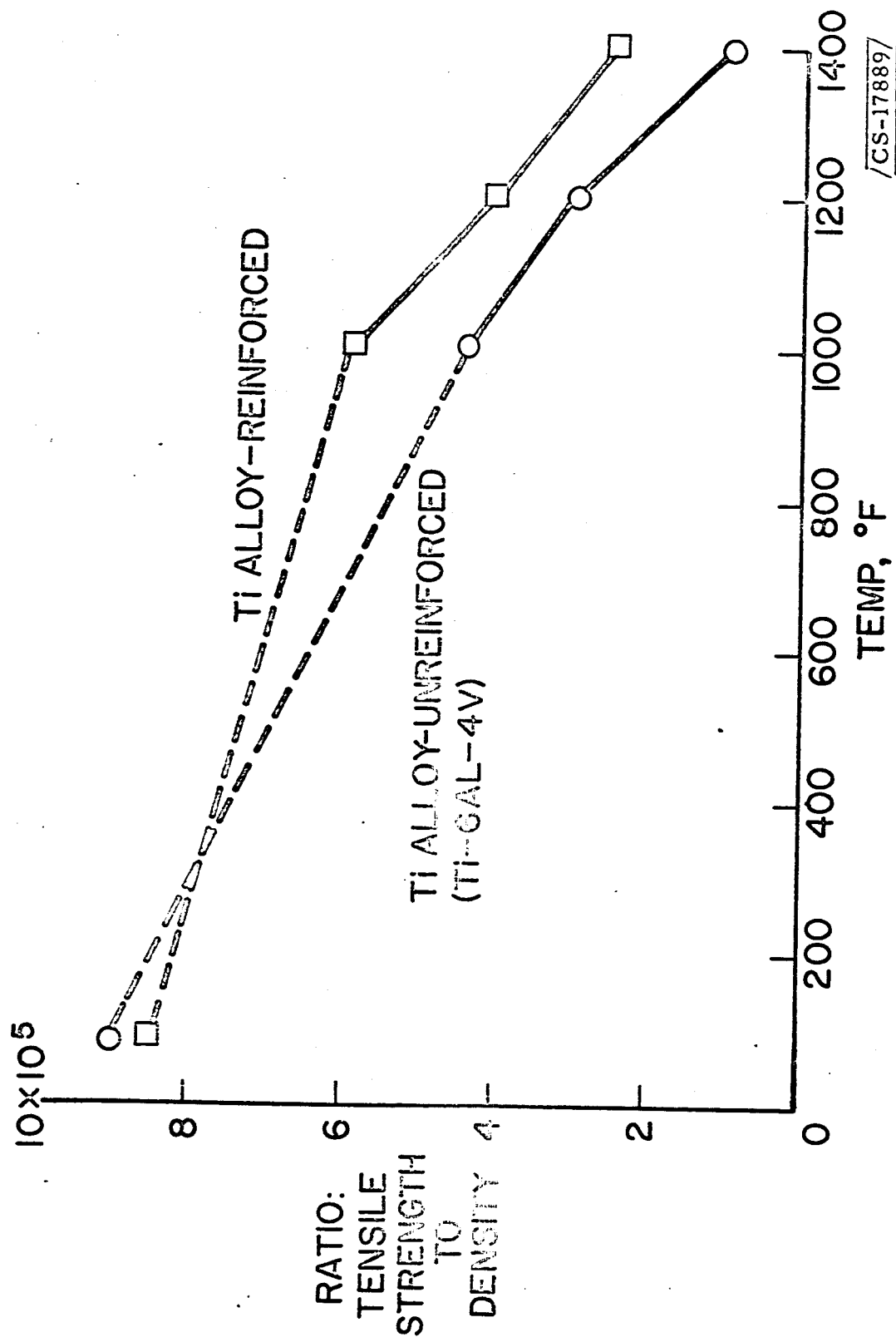


Figure 3. - Tensile strength-density ratio versus temperature for unreinforced titanium alloy and alloy reinforced with 20 volume percent molybdenum fiber.

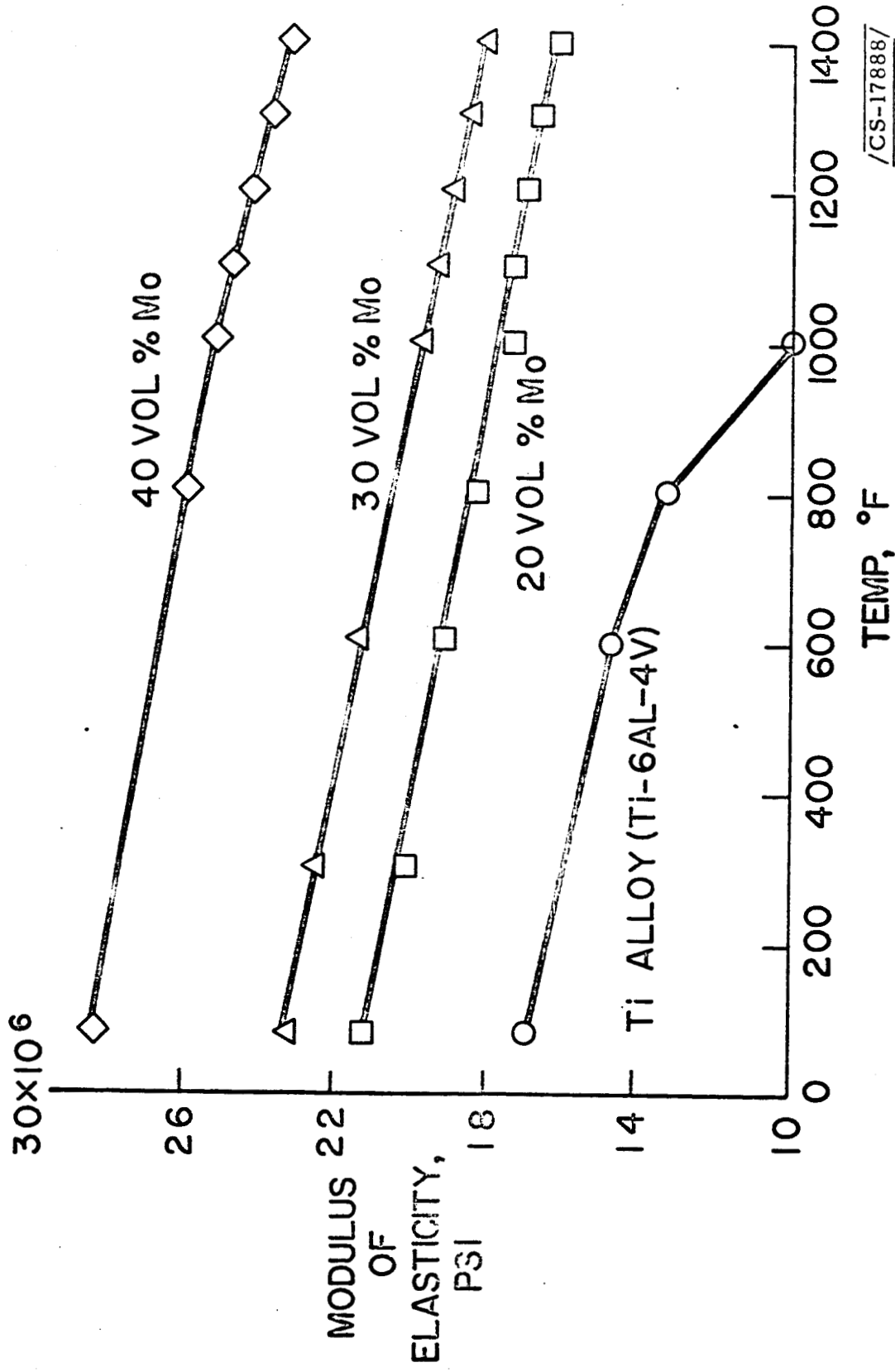


Figure 4. - Modulus of elasticity versus temperature for unreinforced titanium alloy and alloy reinforced with 20, 30, and 40 volume percent molybdenum fiber.

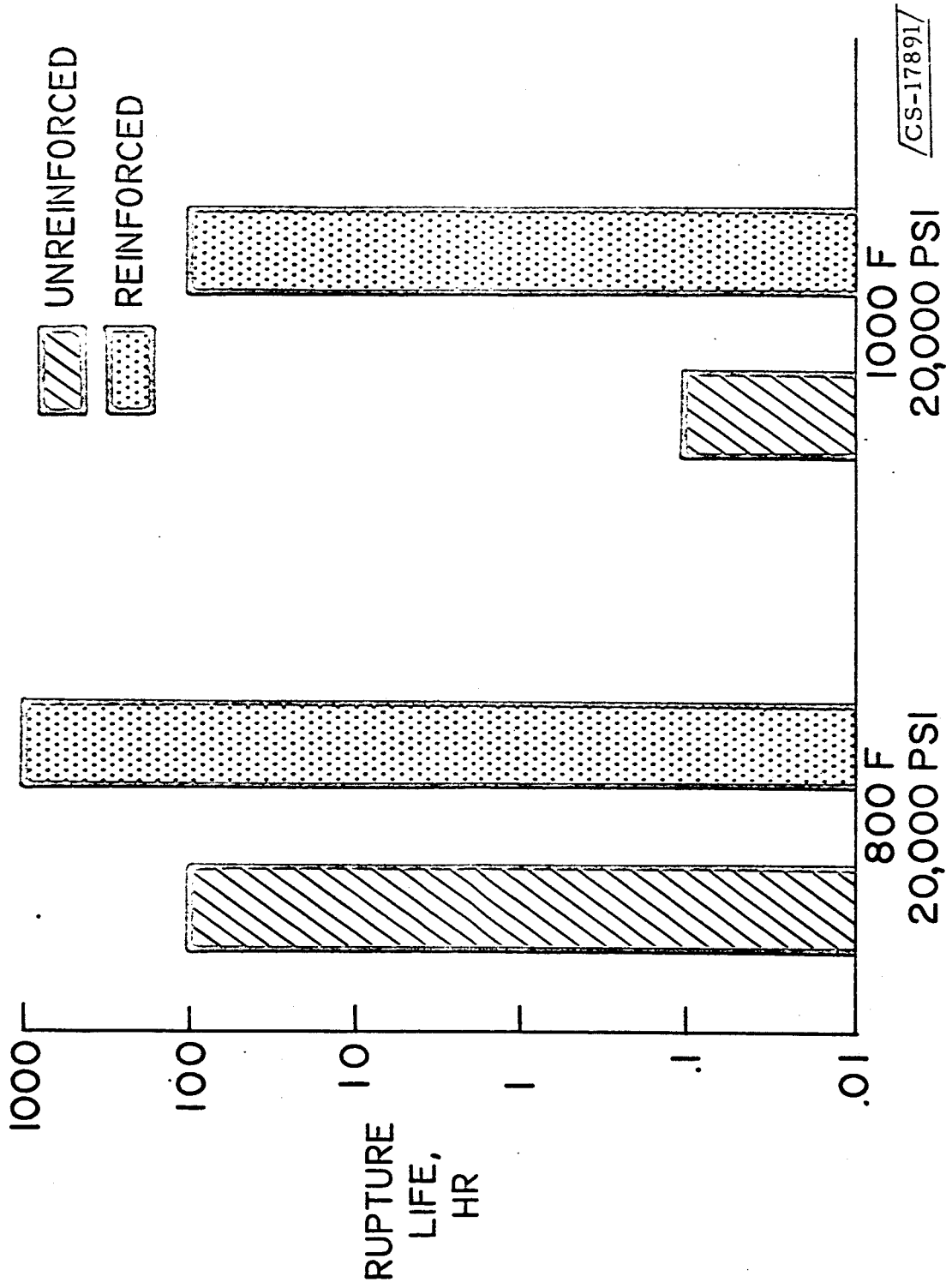


Figure 5. - Comparison of stress rupture life at 800 and 1000 F of unreinforced unalloyed titanium and unalloyed titanium reinforced with 10 volume percent molybdenum fiber.

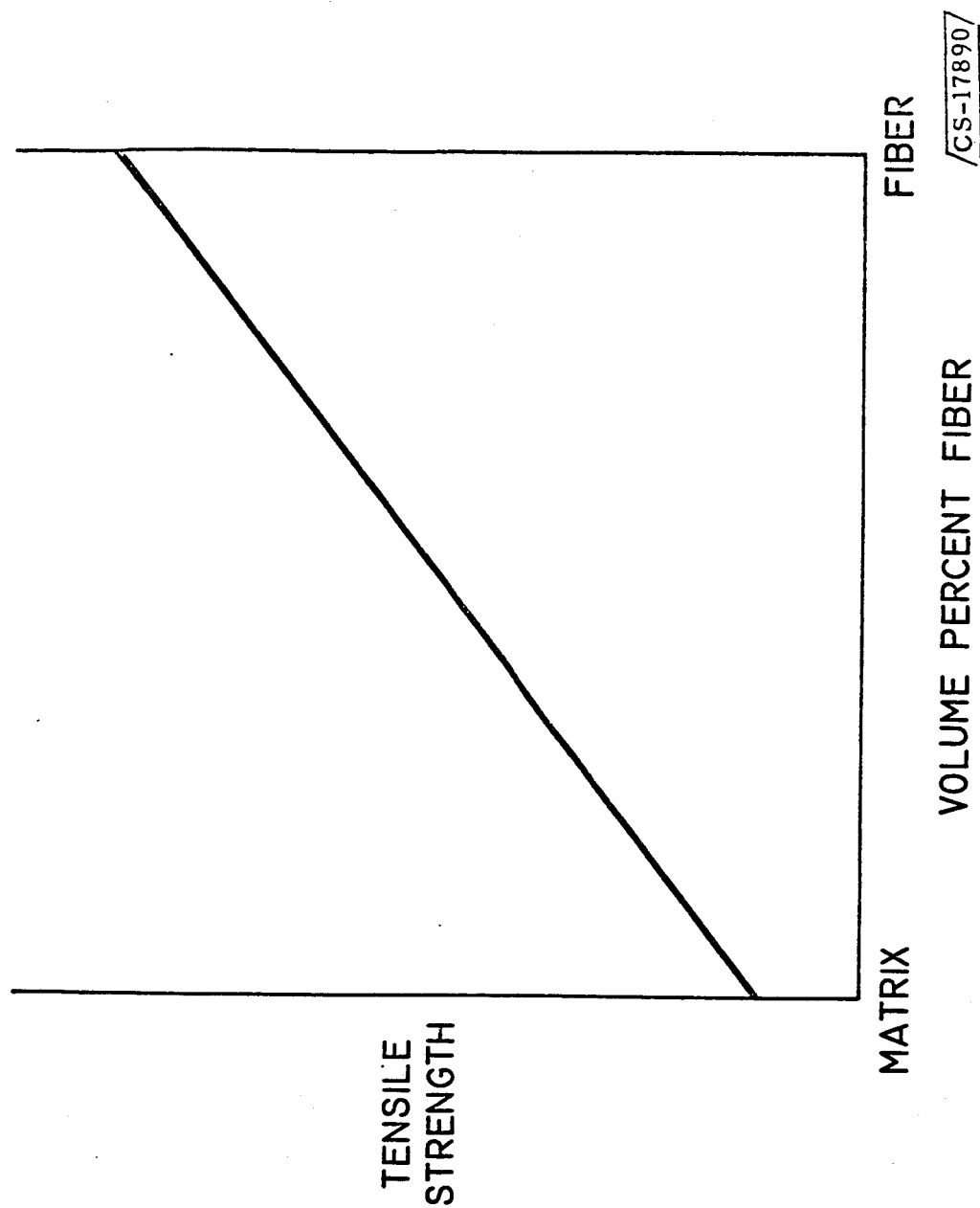


Figure 6. - Schematic tensile strength-composition curve for fiber reinforced composites containing two constituents.

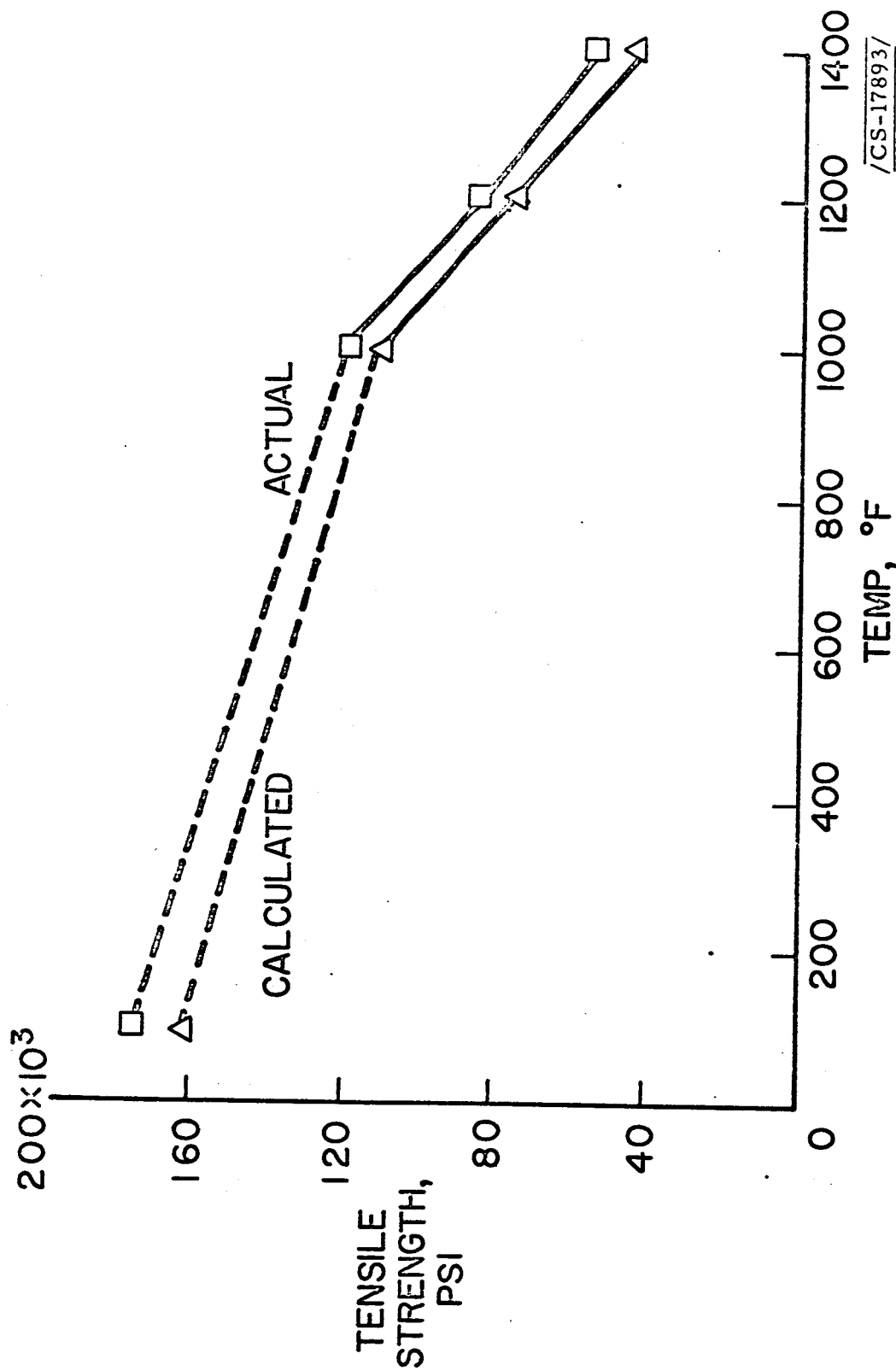


Figure 7. - Comparison of calculated and actual tensile strength of Ti-6Al-4V alloy reinforced with 20 volume percent molybdenum fiber.

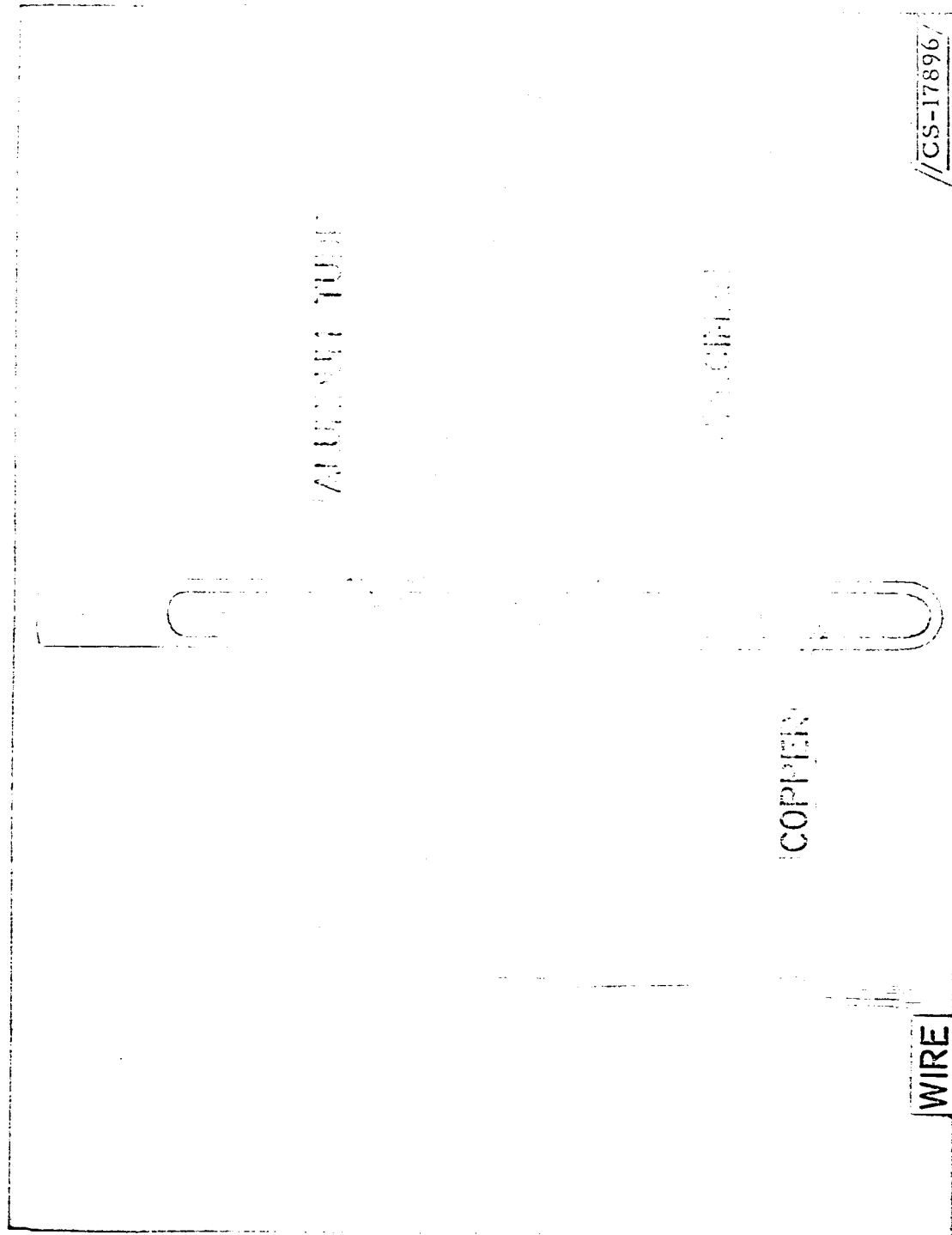
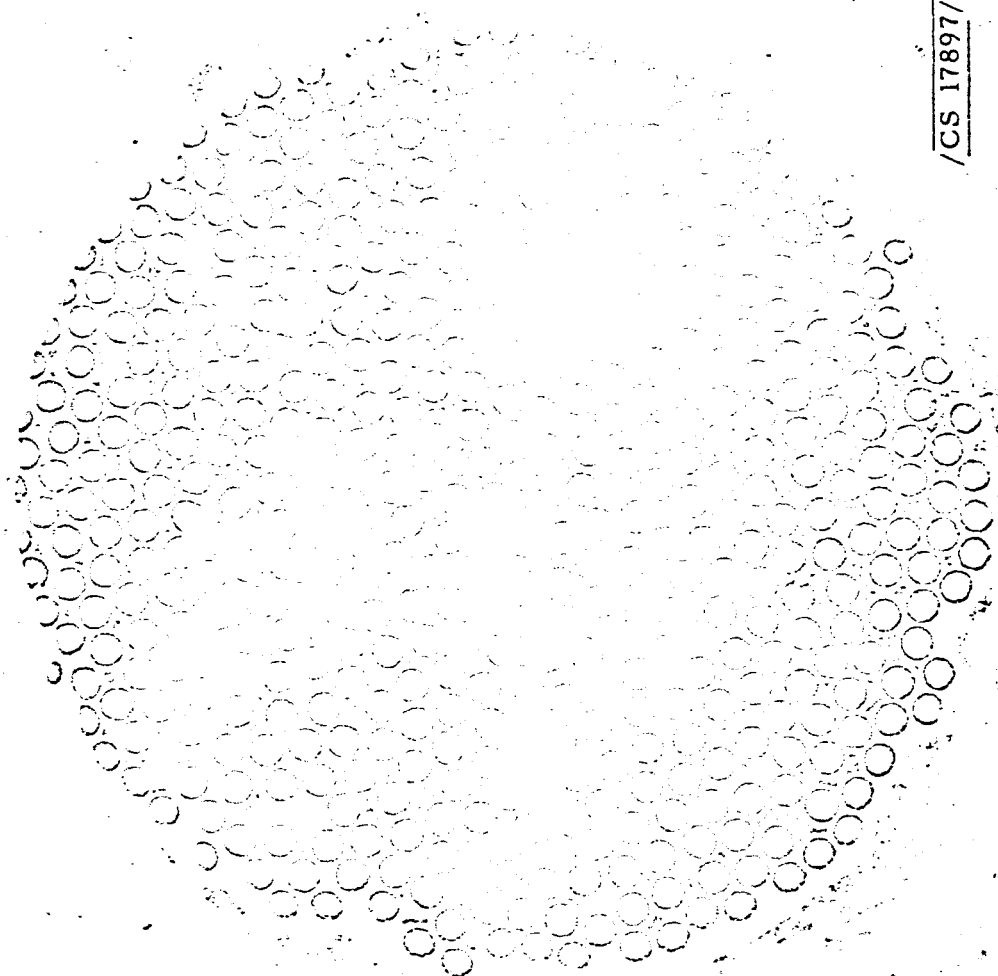


Figure 8. - Steps involved in producing tungsten-copper composites.



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Figure 9. - Transverse section of tungsten reinforced copper composite contain in 483 - 5 mil tungsten wires. Unetched, X50.

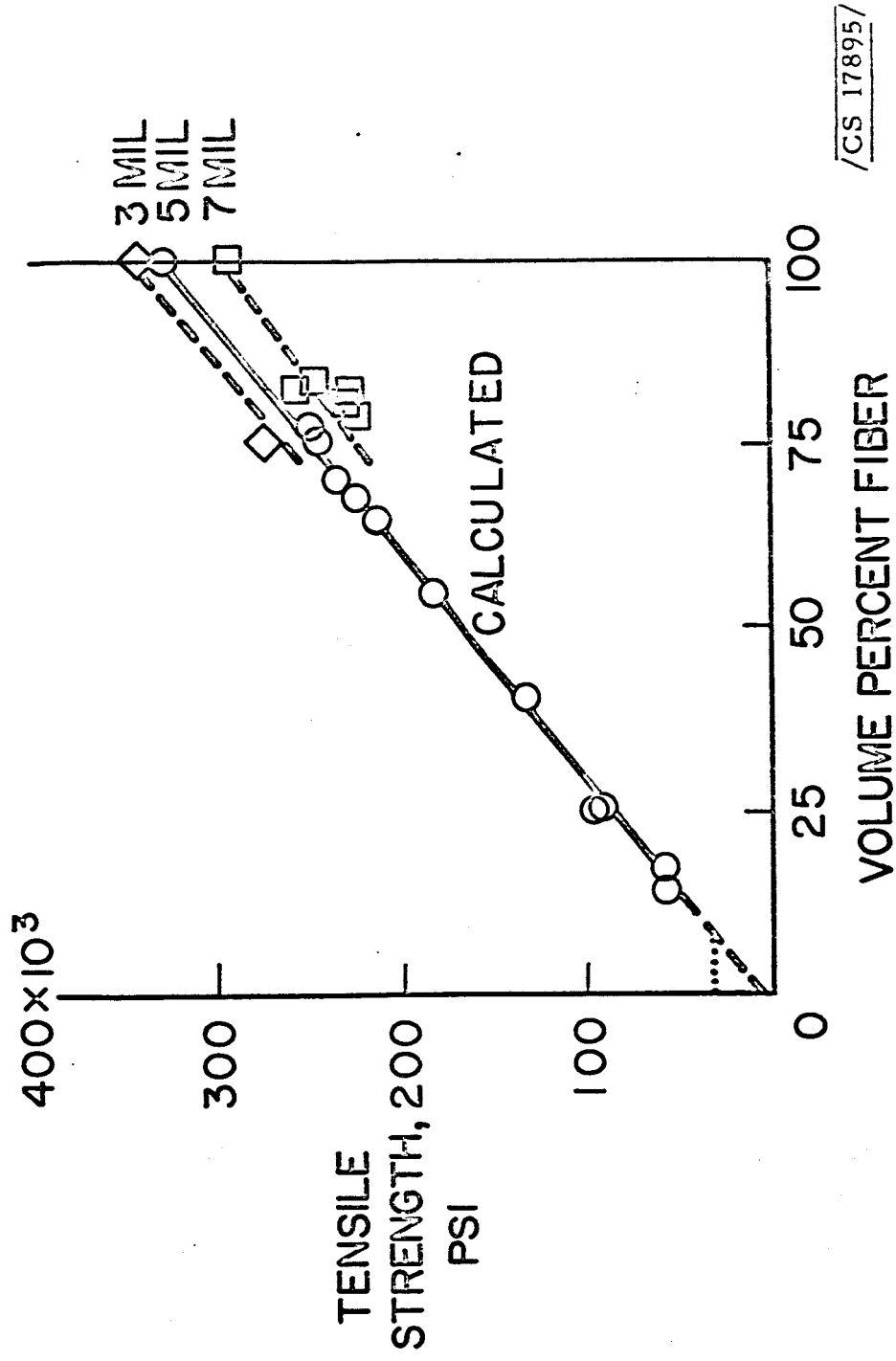
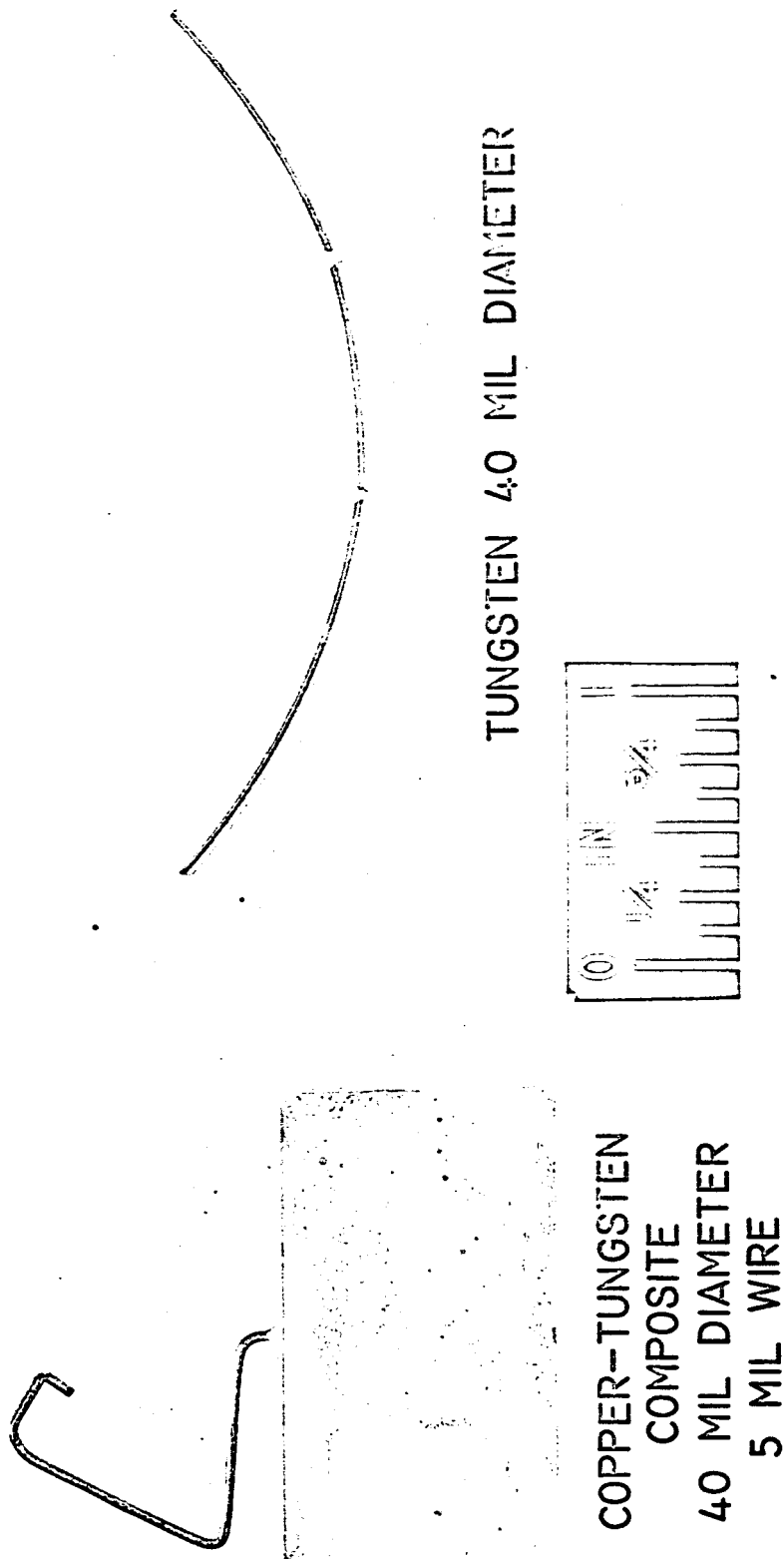


Figure 10. - Comparison of calculated and observed tensile strength of composites containing continuous tungsten fibers in copper.

/CS 17895/



COPPER-TUNGSTEN
COMPOSITE
40 MIL DIAMETER
5 MIL WIRE

TUNGSTEN 40 MIL DIAMETER

/CS-17898/

Figure 11. - Apparent bend ductility of tungsten-copper composite compared to bulk tungsten of same diameter.

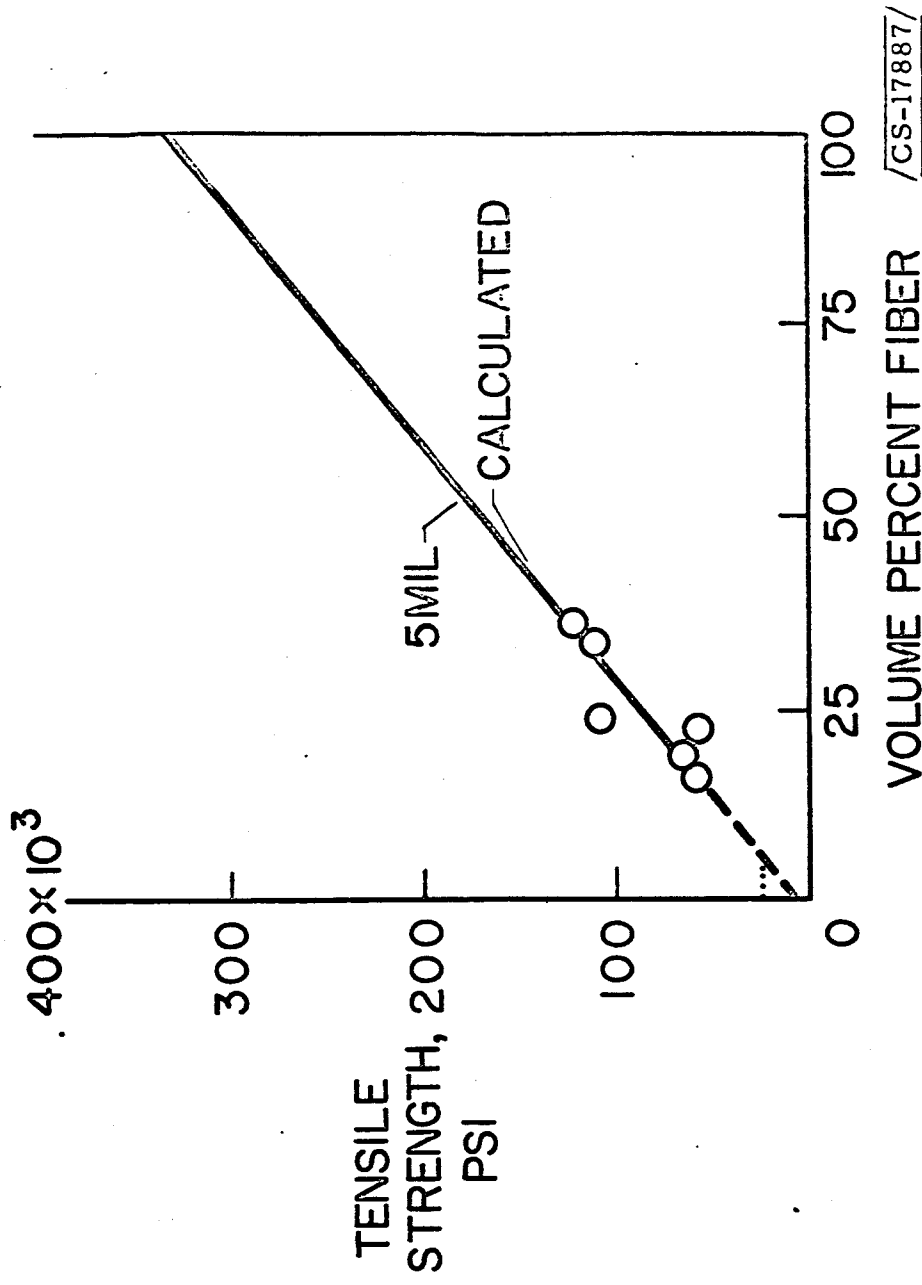


Figure 12. - Comparison of calculated and observed tensile strength of composites containing discontinuous tungsten fibers in copper.

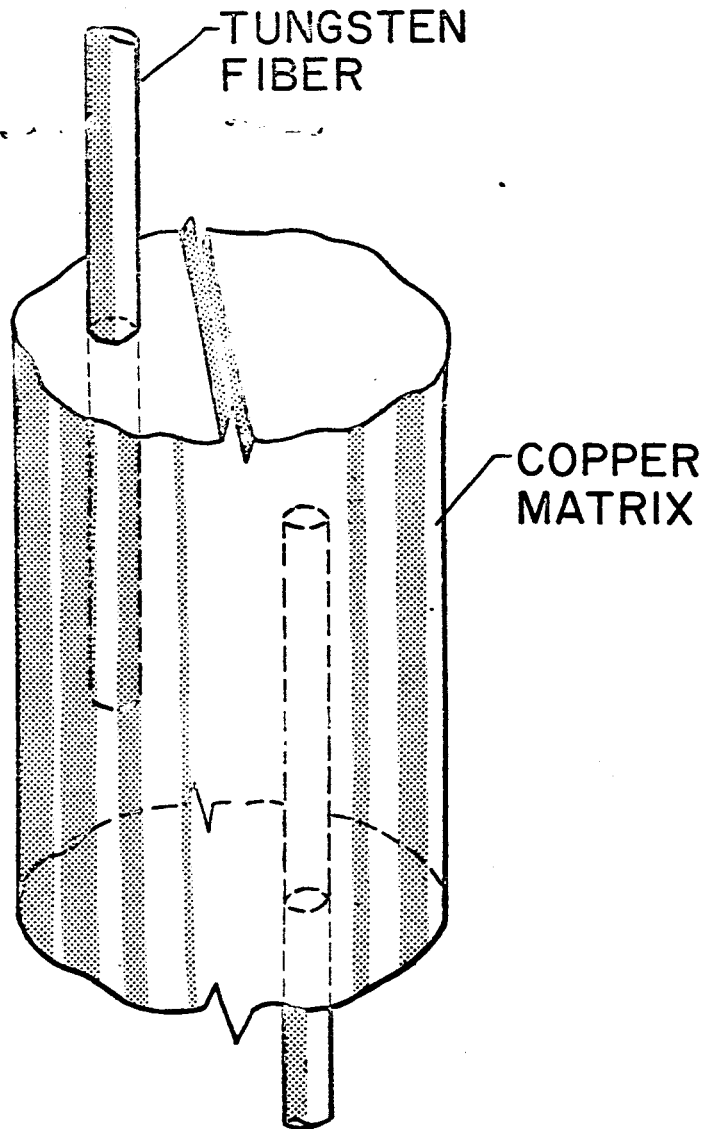


Figure 13. - Schematic loading diagram for an idealized situation.